Low Dose Rate Proton Irradiation of Quartz Crystal Resonators

15 January 1998

Prepared by

R. KOGA, M. D. LOOPER, and S. D. PINKERTON Technology Operations The Aerospace Corporation

and

W. J. STAPOR and P. T. McDONALD NRL Washington, DC 22091

Prepared for

SPACE AND MISSILE SYSTEMS CENTER AIR FORCE MATERIEL COMMAND 2430 E. El Segundo Boulevard Los Angeles Air Force Base, CA 90245 19980605 11(

Engineering and Technology Group



APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED

This report was submitted by The Aerospace Corporation, El Segundo, CA 90245-4691, under Contract No. F04701-93-C-0094 with the Space and Missile Systems Center, 2430 E. El Segundo Blvd., Suite 6037, Los Angeles AFB, CA 90245-4687. It was reviewed and approved for The Aerospace Corporation by A. B. Christensen, Principal Director, Space and Environment Technology Center. Maj. J. W. Cole was the project officer for the Mission-Oriented Investigation and Experimentation Program (MOIE) program.

This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

J. W. Cole, Maj. USAF

SMC/AXES

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Washington Headquarters Services, Directorate for Information operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

AGENCY USE ONLY (Leave blank)	2. REPORT DATE 15 January 1998	3. REPORT	TYPE AND DATES COVERED
4. TITLE AND SUBTITLE Low Dose Rate Proton Irradiation of Quartz Crystal Resonators			5. FUNDING NUMBERS
6. AUTHOR(S) Koga, R., Looper, M.D., Pinkerton, Corporation; Stapor, W.J., McDona	F04701-93-C-0094		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) The Aerospace Corporation			8. PERFORMING ORGANIZATION REPORT NUMBER
Technology Operations El Segundo, CA 90245-4691			TR-96(8570)-3
9. SPONSORING/MONITORING AGENCY NAME Space and Missile Systems Center	E(S) AND ADDRESS(ES)		10. SPONSORING/MONITORING AGENCY REPORT NUMBER
Air Force Materiel Command 2430 E. El Segundo Blvd. Los Angeles Air Force Base, CA 90245			SMC-TR-98-14
11. SUPPLEMENTARY NOTES			
12a. DISTRIBUTION/AVAILABILITY STATEMENT			12b. DISTRIBUTION CODE
Approved for public release; distrib	oution unlimited		

13. ABSTRACT (Maximum 200 words)

Quartz crystal resonators were systematically irradiated with 65 MeV protons to characterize low dose rate radiation-induced degradation. Results indicate: (1) test samples that exhibit large frequency shifts during testing tend to show large frequency shifts prior to irradiation, or during off-irradiation periods; (2) for radiation-sensitive samples, short-term effects seem to decrease after each irradiation on/off cycle (moreover, those devices in which radiation effects do not decrease after a few cycles are not very sensitive); (3) the fabrication process may be an important determinant of susceptibility to low dose radiation-induced degradation; and (4) total-dose effects may be sublinear.

14. SUBJECT TERMS	Quartz resonator of	Quartz resonator degradation	
Heavy ion irradiation effe	ects Radiation effects		12
Proton irradiation effects	South Atlantic An	omaly	16. PRICE CODE
Quartz crystal degradatio	n Space-borne cryst	als	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT

LOW DOSE RATE PROTON IRRADIATION OF QUARTZ CRYSTAL RESONATORS

R. Koga[†], M.D. Looper[†], S.D. Pinkerton[†], W.J. Stapor*, and P.T. McDonald*

† The Aerospace Corporation, El Segundo CA 90245

* NRL, Washington DC 22091

Abstract

Quartz crystal resonators were systematically irradiated with 65 MeV protons to characterize low dose rate radiation-induced degradation. Results indicate: (1) test samples that exhibit large frequency shifts during testing tend to show large frequency shifts prior to irradiation, or during off-irradiation periods; (2) for radiation-sensitive samples, short-term effects seem to decrease after each irradiation on/off cycle (moreover, those devices in which radiation effects do not decrease after a few cycles are not very sensitive); (3) the fabrication process may be an important determinant of susceptibility to low dose radiation-induced degradation; and (4) total-dose effects may be sublinear.

I INTRODUCTION

Piezoelectric crystals are often used to stabilize the frequency of resonator circuits. Because quartz crystals provide negligible mechanical loss and high stability (large Q, quality factor), they have been utilized extensively in providing stable frequency standards. As a complete frequency generating system, crystal oscillators (XO) come in various forms: VCXO (voltage controlled crystal oscillator), TCXO (temperature compensated crystal oscillator), and OCXO (oven controlled crystal oscillator). Each quartz crystal die is "cut" from quartz stock along a special crystal axis for specific applications. For example, the SC (stress-compensated) cut provides improved temperature stability over the traditional AT-cut [1]. Some crystals (such as BVA) are only capacitively coupled to the resonator electronics and are therefore electrodeless [2], whereas others have electrodes adhered to the crystal.

Prolonged use of a quartz crystal results in a gradual shift in frequency. This shift is caused by both physical and chemical changes within the crystal; frequency can also be affected by the adhered electrodes. The change in frequency, $\Delta y(t)$, can be approximated by: $\Delta y(t) = \alpha t$, where α is on the order of 1 x 10⁻¹¹/day and t is time [3]. More generally, $\Delta y(t) = A \ln (Bt + 1)$ where A and B are appropriate constants [4].

Much larger frequency alterations are experienced by crystals when irradiated by natural radiation in space [5]. Numerous studies on the radiation susceptibilities of crystal material and crystal resonators have therefore been conducted [6-10].

Effects of radiation on the properties of quartz have been studied using various techniques over the years [11-13]. In order to decrease radiation effects, manufacturers often subject crystals to an electrodiffusion (sweeping) process. This technique consists of applying an electric field along a crystal axis while maintaining the sample temperature at about 500°C. Because it is difficult to predict frequency stability

characteristics of crystal oscillators from the properties of the crystal itself (such as the density of defect centers and the concentration of impurities), the quartz crystals in resonators (oscillators) are often irradiated directly to observe radiation effects. Within the last dozen years many radiation effects have been observed and elucidated. Some of them are:

- 1) For many resonators, application (preconditioning) with about 20 krad of Co⁶⁰ gamma-ray radiation appears to decrease further frequency degradation due to subsequent irradiation [5]. Therefore, quartz resonators used in space applications are usually preconditioned.
- 2) The effect of radiation on resonators is not linear and extrapolation of high dose rate (well above a few hundred rad(Si)/min) effects to lower dose rates (well below a few rad(Si)/min) is not reliable [6,7].
- 3) High dose rate sensitivities are more strongly governed by the impurity levels of the raw crystal material [8]. Therefore, the study of impurities may contribute to understanding high dose rate effects.
- 4) There are indications that the sensitivity of resonators to low dose rate irradiation may be affected by variations in manufacturing processes. Variations can arise in the process of adhering electrodes, as well as the crystal manufacturing process itself. As a consequence, lot testing could be useful in characterizing the expected sensitivity of quartz resonators [8].
- 5) Proton irradiation of crystals resonators can result in greater degradation of frequency stability than occurs with gamma rays [8].
- 6) A large change in frequency per rad occurs at low dose rates
- 7) The exact cause of degradation is not well known [5,9].

Because high accuracy time standards are essential in many space applications, it is important to investigate crystal resonator degradation effects due to radiation, especially low dose rate effects in the radiation belt. Here, it is important to study short-term effects as well as the gradual deterioration of crystal resonators over long time intervals. Many spacecraft pass through the South Atlantic Anomaly (SAA) region of the radiation belt where radiation peaks. Encounters with low dose rate protons in the SAA may produce frequency shifts in onboard clock resonators. In applications that require precision clock frequencies, such rapid fluctuations could be deleterious.

Quartz crystals are highly cost-effective relative to atomic standard clocks, hence their use in space is expected to continue. In the following, we describe some recent systematic measurements of the susceptibility of quartz crystals to low dose rate protons.

II TEST DEVICES

The test vehicles for the quartz resonators were FTS1130 oscillators. The FTS1130 is a temperature-controlled, high performance device with a temperature coefficient of less than

Table 1. List of Test Samples with Lot Number and Allan Variance $[(AV \times 10^{-12})]$ is measured at $\tau = 600$ seconds

Lot Number	Serial Number	Frequency (MHz)	Allan Variance (AV)*
9450	1486	10.24	11
9450	1488	10.24	90
9451	1485	10.24	18
9509	1487	10.24	5
9509	1490	10.24	9
E	1489	10.24	3
9450	1478	10.95	NA
9450	1483	10.95	45
9450	1484	10.95	18
9512	1480	10.95	11
9512	1481	10.95	12
E	1479	10.95	3
Ē	1482	10.95	19

* Allan variance for irradiation period

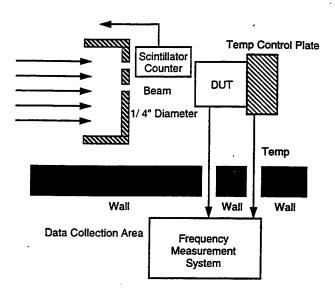


Fig. 1. Proton irradiation test set-up (DUT = device under test)

5 x 10⁻¹²/°C. The tested crystals were third overtone SC-cut crystals in a HC-40 style holder manufactured by Bliley Electronics Company. The crystals were cut from swept quartz and were preconditioned by irradiating the devices to a level of 23.3 krad (dosimetry averaged) over 20 minutes using a Co⁶⁰ source at Raytheon (Sudbury, MA). The crystals were of two frequencies: 10.24 MHz (six units) and 10.95 MHz (seven units). The resonators were electrode type and were spares from actual production lots intended for space applications. The test devices are listed in Table 1 ("Allan variance" is defined in the Results section.)

III EXPERIMENTAL CONDITIONS

To investigate characteristic responses to irradiation with low dose rate protons, we systematically irradiated about a dozen quartz crystal resonators with 65 MeV protons at the University of California Davis cyclotron facility. A scintillator

detector was used to monitor the flux and the fluence of the proton beam, as shown in Fig. 1. The test oscillator was placed in the beam and was compared with a control oscillator that was out of the beam. (Only the crystals were irradiated; the drive electronics were shielded from irradiation.) The test oscillator was attached to a temperature-controlled aluminum plate held to within ±0.2°C. The oscillator frequency was measured by mixing the output frequency against a synthesizer with a 100 Hz offset, as shown in Fig. 2. The frequency synthesizer was referenced to a FTS4050 cesium standard. The output of the mixer (a FTS6102 Beat Box) was measured using HP53132A counters with a 10 second gate time. Recorded measurements included the fractional frequency difference ($\Delta f/f$), temperature, humidity, barometric pressure, event markers, and time interval. (Fractional frequency difference is also termed frequency deviation, frequency shift, or frequency change.)

A proton energy of 65 MeV was chosen as a realistic indicator of effects in the 40-100 MeV range representing the broad peak for LEO (low Earth orbit) protons passing through aluminum with thickness on the order of 1 gm/cm². The quartz blank of the resonator (about 0.3 gm/cm²) absorbs approximately 3% of the energy of each proton. The test bed

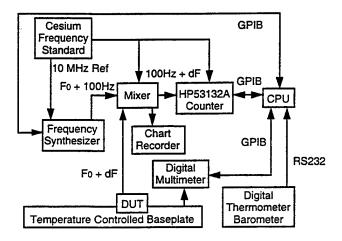


Fig. 2. Data collection systems

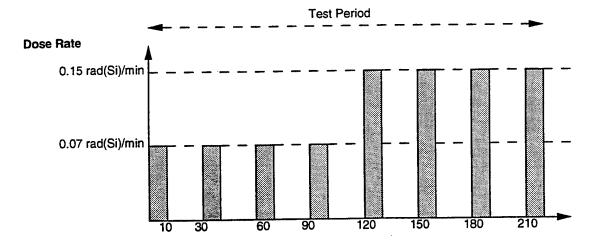


Fig. 3. Schematic illustrating 10-minute proton irradiation periods separated by 20-minute annealing (off-irradiation) intervals

oscillator shielded the crystal with 1 gm/cm² equivalent aluminum. The effective shielding degraded the proton spectrum peak to 57 MeV, which resulted in a 1.5% dose increase at the crystal, relative to the observed scintillator detector dose. The incident doses were adjusted accordingly.

The crystal resonators were irradiated for 10 minutes every 30 minutes while the dose rate was maintained at 0.07 rad(Si)/min (at 8 x 10³ particles/cm²-sec) or 0.15 rad(Si)/min (at 1.7 x 10⁴ particles/cm²-sec), as indicated in Fig. 3. Most of the crystals experienced four cycles at both radiation levels. The 10-minute interval during which the crystal resonators were exposed to the proton beam is called the "irradiation period"; the subsequent 20-minute interval is the "offirradiation period"; and the time interval between the beginning of the first and the end of the last irradiation periods is termed the "test period." For some resonators, measurements were taken during the test periods as well as during pre- and posttest periods. The test bed oscillators were positioned in such a way as to place the crystal in the path of the protons with the quartz blank perpendicular (+Z-axis) to the beam.

The Allan variance of the crystals was measured as a means of characterizing degradation in frequency stability. (The Allan variance is approximately equal to the root-mean-square fluctuation in the fractional frequency [14], and is the defacto standard for characterizing frequency stability.)

The intrinsic frequency fluctuations of the cesium standard used as the primary frequency reference show Allan variance equal to about 3 x 10^{-11} (for a characteristic time interval, $\tau=0.5$ sec) within the observation time periods. During the 10-second observation period, the reference contributed 1 x 10^{-11} units of ($\Delta f/f$) noise. The observed noise in the measured frequency of each oscillator under test is a combination of test device and reference noise.

Three additional tests were performed. The first measured the directional sensitivities of two crystals in the $\pm Z$, $\pm Y$ and $\pm X$ direction. The second test measured the effects of high levels of radiation exposure, 300 krad(Si) and 900krad(Si), on two resonators. The third test assessed the recovery response of one of most radiation sensitive crystals. The response was measured at 9 and 36 hours after the initial irradiation period.

IV TEST RESULTS AND ANALYSIS

The data consisted of frequency deviation ($\Delta f/f$) measurements of the crystal oscillators, which were observed every 10 seconds during the test period. One example of the data obtained is shown in Fig. 4, which illustrates the frequency shift of SN1490 during both the pre-test and test periods (the pre-test period is indicated by negative time values along the x-axis).

A. Segmented Linear Fits

The rate of change of the fractional frequency difference ($\Delta f/f$) was approximated by a global least square fit, as shown in Fig. 5 for SN1481. The rate of change of $\Delta f/f$ is:

$$\Delta y/\Delta t = \frac{(\Delta f/f)_{i+1} - (\Delta f/f)_{i}}{\Delta t}$$
 (1)

where $\Delta t = t_{i+1} - t_i$.

Because $\Delta f \ll f$ for all crystal oscillators, the reference frequency of each oscillator was chosen as the nominal frequency at the start of the test period. Within a single 10minute irradiation period there were 60 data points (one every 10 seconds). Data were averaged over two-minute intervals, as shown in Fig. 5, and linear fits were calculated based on the two-minute averaged values of $\Delta f/f$. One of the constraints of the plotting routine is that the linear fit lines be tied from one segment to another; in this sense, the fit is global. The error bars were calculated using the data points within the adjacent segments. Because the fit is global, the error bars tend to spread wider than the plotted data points. During the offirradiation period, least-square fits were carried out for each 20minute interval in a similar fashion. The graphs show the spread of the data points, as well as the trend of the frequency drifts in each segment.

The rate of change of $\Delta f/f$ (or equivalently the fractional rates of change of f) during the irradiation and off-irradiation periods are compared for device SN1481 in Fig. 6. As expected, the rate of change is larger and exhibits greater variability during irradiation. For moderately unstable samples, a trend is apparent in the data. For example, as shown in Fig. 6 for SN1481, there is a noticeable jump from the last data

Table 2. Minimum to maximum range of fractional rate of change of frequency over about 240 minutes of test period

Serial Number	During off-irradiation period (10 ⁻¹² /sec)	During irradiation period (10 ⁻¹² /sec)
1479	.01	.04
1480	.03	.07`
1481	.04	.06
1482	.02	.07
1483	.05	.4
1484	.04	.06
1485	.05	.1
1486	.02	.07
1487	.01	.02
1488	.05	.6
1489	.01	.03
1490	.02	.04

point taken at 0.07 rad(Si)/min to the first data point at 0.15 rad(Si)/min. Each subsequent data point exhibits even greater change, until saturation is apparently reached. For very unstable devices, such as SN1488, this pattern – jump, followed by subsequent saturation – is very pronounced. (Data for SN1488 are not shown due to space limitations.) For stable device, such as SN1479, the data points vary, but the magnitude of changes is small and there is no obvious trend.

Although space limitations preclude presenting results for the other samples, variability in the "fractional rate of change of f" is summarized in Table 2. The range between the highest and lowest values of the fractional rates of change of f is calculated for both the irradiation and off-irradiation periods. As can be seen, samples that exhibited large variations during the irradiation period (e.g., SN1483) also displayed relatively large variation during the off-irradiation periods.

B. Allan Variances

Another index of frequency stability is provided by the Allan variance of the frequency shifts, as shown in Figs. 7 through 10. (The Allan variance is normalized to the nominal frequency of the device for ease in comparing different devices.) The variances during the test period are plotted in Fig. 7 for oscillator crystal SN1481. In order to ascertain whether or not there are differences in the Allan variance for the two rates of irradiation (0.07 rad/min and 0.15 rad/min), additional calculations were performed. The variances for the data obtained during irradiation at the two dose rates are shown in Fig. 8. Data from the first half of the test period were used for the Allan variance calculations at the lower dose rate (0.07 rad/min), whereas data from the second half of the test period were used for the higher dose rate (0.15 rad/min) calculations. As is illustrated in the figure for SN1481, for most samples the Allan variances were greater at the high dose rate than at the lower rate. Only three exceptions were noted (samples SN1483, SN1484, and SN1488), all of which were from lot 9450. However, samples SN1486 and SN1478 from lot 9450 exhibited the "standard" tendency (greater Allan variances at higher doses). Thus, wide device-to-device variance has been observed for this lot.

The Allan variances calculated for the pre-test and test periods, respectively, are shown in Figs. 9 and 10 for SN1490, which represents a typical case. Space limitations prevent us from presenting figures for all test devices; however, Table 1

provides a summary of the Allan variances exhibited during the irradiation period (e.g., see Fig. 7) at $\tau = 600$ seconds.

C. Comparison with Pre- and Post-test Data

In this section we compare the frequency variations observed during the test period with those observed prior to, and following, this period. Since journal space is limited, we will summarize the test results with only a small number of figures. For samples SN1484, SN1487, and SN1490, the frequency was increasing prior to the test period, and continued to increase at similar (or slightly higher) rates during the test period, as shown, for example, in Fig. 4 for SN1490. For SN1480, the increasing frequency of the pre-test period was reversed during the test period. For SN1479, the frequency increased at a higher rate after the irradiation. For SN1486, the frequency decreased during the irradiation period; however, there was a tendency for the frequency to increase during the posttest period. In summary, during the test period, the frequency tended to decrease prominently (SN1479, SN1480, and SN1486), remain the same (SN1487), or increase slightly (SN1484 and SN1490); for other devices we do not have sufficient data to permit comparison. Moreover, the data are not sufficient to determine long range frequency trends after the test period; further study is needed to detect consistent tendencies in this area.

D. Directional Sensitivity

The relationship between the frequency shift and the direction of the beam with respect to the orientation of the crystal axes was investigated. Detailed test results were reported at the European Forum on Time and Frequency, held in March 1996 [14]; only a summary is provided here. As noted above, most exposures were conducted with the beam oriented along the normal (+Z-axis) direction of the crystal. However, for comparison purposes, several data points were also collected with the beam impinging at different directions. Three directions ($\pm Y$ and +X) parallel to the crystal surface were chosen; the other orientation (-X direction) was observed by the oscillator electronics and was not measured. The results indicate that the sensitivity along these axes are very similar. Because there was no preferred direction for amplified sensitivity, results (such as those reported here) obtained from a particular direction have general applicability to the space radiation environment.

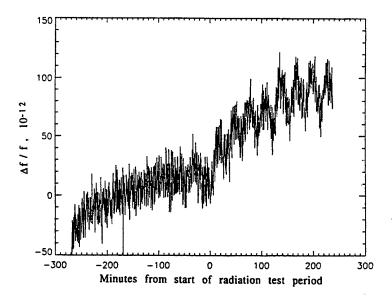


Fig. 4. Frequency shifts of SN1490 resonator during pre-test and test periods

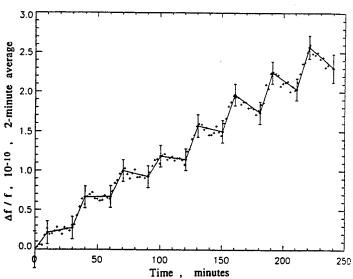


Fig. 5. Frequency shifts of SN1481 resonator during test period

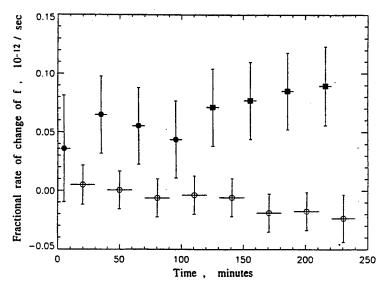


Fig. 6. Rate of change of Δf/f for SN1481 during irradiation (closed circles) and off-irradiation (open circles) periods. The first four data points were collected at a dose rate of 0.07 rad(Si)/min, and the second four at 0.15 rad(Si)/min

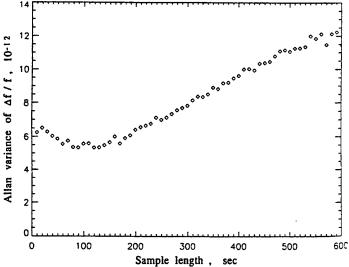


Fig. 7. Allan variance of SN1481 resonator during test period

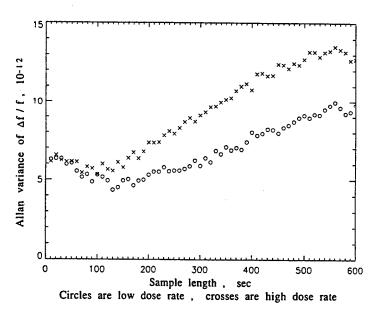


Fig. 8. Allan variance of SN1481 resonator during low (0.07 rad(Si)/min) and high (0.15 rad(Si)/min) dose rate irradiation

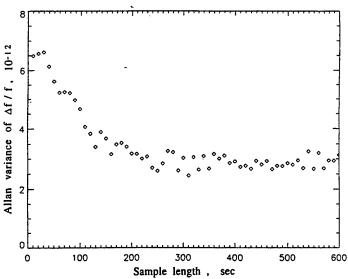


Fig. 9. Allan variance of SN1490 resonator during pre-test period

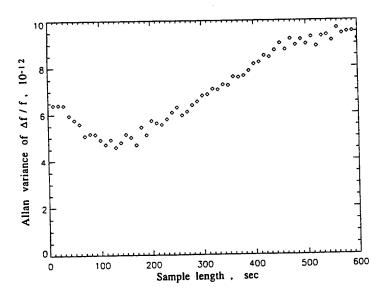


Fig. 10. Allan variance of SN1490 resonator during test period

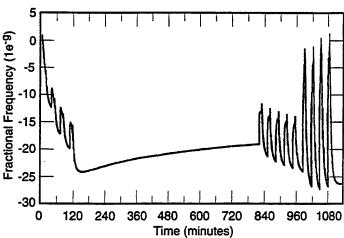


Fig. 11. Total dose sensitivity of SN1489 resonator (exposures: 3 x 20K, 5 x 40K, 4 x 160K)

E. Total Dose Sensitivities

Two devices, SN1490 and SN1489, were irradiated to high total dose levels of 300 krad(Si) and 900 krad(Si), respectively. (Frequency variations due to exposure are reported in the proceedings of the European Forum on Time and Frequency; only the net results of the irradiation are described here.) Figure 11 shows the 900 krad(Si) total dose effects on oscillator SN1489 (the results for SN1490 were similar). The crystals were irradiated in several increments of 20 krad to 160 krad, separated by a time interval of about ten-hours during which no irradiation occurred. The net sensitivities of the two oscillators to total dose irradiation were 2 x 10⁻¹⁴/rad and 3 x 10-14/rad, for SN1490 and SN1489, respectively. These sensitivities were three orders of magnitude lower than the low dose rate sensitivities for the same resonators. This suggests that there is a tendency for sensitivity to decrease with increasing dose.

F. Recovery Responses of Sensitive Crystal

During the test period, SN1488 was irradiated a total of 10 times. The results showed that this device was one of the most sensitive ones (see Tables 1 and 2). Following the irradiation period the resonator was left unperturbed for 9 hours. It was then subjected to a low dose rate exposure of 0.7 rad. The $\Delta f/f$ response of 12.9 x $10^{-11}/r$ rad was less than the value for any previous 0.7 rad exposure. The resonator was then left for 36 hours and exposed again to 0.7 rad and 1.5 rad in succession. The responses were 8.6 x $10^{-11}/r$ rad and 5.3 x $10^{-11}/r$ rad, both less than for any of the original 10 exposures.

V DISCUSSION

Further implications of the above findings are discussed in detail below.

A. Rates of Frequency Change during Irradiation Periods

Rates of frequency change (within the 10-minute time interval) were obtained (for example, see Fig. 4). In some cases, the magnitude of the rate of change decreased with each successive irradiation period at the 0.07 rad/min dose rate, and there was a large jump in the magnitude of the rate of change for the first irradiation period at the 0.15 rad/min rate. However, the magnitude of the rate of change decreased again with each successive irradiation period at the higher dose rate. The samples for which this trend was clearly observed (e.g., SN1483 and SN1488), happened to be highly susceptible to irradiation. Devices for which the trend was not easily detected (or for which there was no such trend), were less sensitive to radiation. In contrast, in a previous journal article, Suter, Maurer, and Kinnison reported that their test device(s) displayed a tendency toward reduced rates of change with successive irradiations [5]. However, it appears that their test sample size was small; it is possible that the limited number of devices they tested all displayed a trend toward reduced rates of change. In contrast, the number of test samples utilized in the present investigation was large enough to include devices with varying test results. However, one of most sensitive devices (SN1488) did follow the trend suggested by Suter et al.

The rates of frequency change during irradiation, $\Delta y/\Delta t$, varied. But, for any single resonator, observed variances were

usually confined to a narrow region. For SN1484, for example, $\Delta y/\Delta t$ varied from 0.05 x $10^{-12}/\text{sec}$ to 0.12 x $10^{-12}/\text{sec}$. Therefore, in some cases, a trend for $\Delta y/\Delta t$ may be established within about half the duration of the present test (about 240 minutes).

The rates of frequency change observed during the offirradiation periods (20-minute time intervals) tended to be smaller than during the irradiation period (see Table 2). In addition, variation in the rate of change during the offirradiation period was small. Therefore, the annealing process appears not to be drastically affected by the dose rates within the irradiation periods.

B. Allan Variance

The Allan variance displayed frequency changes at different characteristic times. For sample times of less than 100 seconds, the frequency variance was often caused by short term noise, such as inherent crystal instability, noise generated in the oscillator system, or noise associated with the test equipment. The greater variances observed at higher time values may be attributed to irradiation effects or to noise generated within the crystal.

For device SN1484, the Allan variance increased during irradiation. However, large Allan variances were also evident in the pre-test data of the same sample (unlike SN1490, which displayed different characteristics, as shown in Figs. 9 and 10). Therefore, we may conclude that radiation effects, as well as other factors, contributed to the large Allan variance of this sample. As noted earlier, the Allan variances of crystals SN1483, SN1484, and SN1488 were larger at 0.07 than at 0.15 rad (Si)/min. Therefore, we suspect that these samples tended toward large Allan variances prior to irradiation. Even after sweeping the crystal and preconditioning with Co⁶⁰ gamma rays, the crystal resonators may have not been optimally stabilized.

The Allan variances obtained for SN1479, SN1480, SN1486, and SN1490 during the pre- and post-test periods seem to indicate that radiation is the main cause of the increase in the Allan variance for these oscillators.

C. Recovery Responses of Sensitive Crystal

The recovery response of one of the most sensitive crystals, SN1488, tended to improve with successive exposures to radiation. Because the exposure steps used in the tests were different from the periodic exposures, further investigation (with more test samples) is needed to state whether or not this method could be applied to improve the sensitivity of crystals in general.

VI CONCLUSIONS

The results of testing 10.24 MHz and 10.95 MHz third overtone SC-cut quartz crystals confirm some of the earlier studies by Norton et al. and Suter et al. on fifth overtone AT-cut, and SC-cut crystals, respectively [6-8]. Moreover, we have identified additional characteristics of crystal oscillator responses to low dose rate irradiation. The results of the present analyses suggest the following conclusions:

1) The radiation susceptibility of crystals varies from one sample to the next.

2) Two of the samples from lot number 9450 (SN1483 and 1488) exhibited unusually large Allan variances, as shown in Table 1. The same two devices display large frequency variations, especially during the irradiation period (see Table 2). It is important to note that both methods of analysis (involving calculation of the fractional rate of change of frequency, or the Allan variance, as shown in Figs. 6 and 7, respectively) can be used to aid quantitative evaluation of stability.

The existing data show that a few devices in Lot 9450 have much greater instability of frequency than those from other lots. This suggests that the fabrication process may be an important determinant of a crystal's susceptibility to low dose rate radiation-induced degradation. However, further testing with more samples is needed to permit a more definitive statement to be made on the subject of lot-dependent radiation effects.

- 3) Test samples that exhibit large frequency shifts during the test period appear to show large frequency shifts prior to irradiation, as well as during off-irradiation periods (e.g., SN1483 and 1488 in Table 2). However, the converse is not true. Devices such as SN1482 and 1486 display a relatively large instability during the irradiation period, but not during the off-irradiation period. This demonstrates that the low dose rate proton test used here to simulate the radiation environment can indeed reveal differences in the radiation sensitivity of various resonators.
- 4) For some highly unstable test samples, such as SN1488, short-term radiation effects appear to decrease after each irradiation on/off cycle. However, the magnitude of reduction decreases also. In other words, the improvement does not continue at the same rate after each irradiation on/off cycle. In devices with moderate instability such as SN1481 (see Fig. 6), the fractional rate of change of frequency may actually increase at a dose rate of 0.15 rad (Si)/min and decrease at 0.07 rad (Si)/min after each irradiation on/off cycle. Also, for SN1481, it is possible to detect a trend in the data points. For device samples that are very insensitive to radiation, we often did not observe a specific trend. In other words, those devices in which the fractional rate of change of frequency does not change after the first couple of irradiation on/off cycles (SN1479, 1487, 1489, and 1490) are not very sensitive to irradiation.
- 5) The present data show that there was no strong preferred polarity in the frequency change.
- 6) Additional tests with doses up to about 1 Mrad indicated a non-linear effect of total dose with low dose radiation. The total dose sensitivity is about 2 x 10⁻¹⁴/rad at about 1 Mrad, which is orders of magnitude lower than the sensitivity measured at lower doses (about 10 rad). Thus, for this example total dose effects are sublinear, as suggested by Fig. 11.

In the crystal resonator samples evaluated here, frequency variation due to radiation effects exceeded that due to other factors. It is seldom easy to predict how, and to what extent, radiation-induced resonator degradation will affect system performance in a particular application. Therefore, it seems

prudent to conduct irradiation tests of candidate crystal resonators for high precision applications, and to remove obviously vulnerable ones (which do not meet a priori standards) from the list of usable samples. (Sample SN1488 of the current set may belong to this category.) For those devices eventually used in actual applications, irradiation test records should be retained as a possible aid in predicting the expected frequency variation in space.

VII REFERENCES

- EerNisse, E.P., "Rotated X-cut Quartz Resonators for High Temperature Applications," Proceedings of the 32nd Annual Symposium on Frequency Control, 255-269, 1978.
- Besson, R., "A New Piezoelectric Resonator Design," Proceedings of the 30th Annual Symposium on Frequency Control, 78-83, 1976.
- Rueger, L., J.R. Norton, and P.T. Lasewicz, "Long Term Performance of Precision Crystal Oscillators in a Near-Earth Orbital Environment," Proceedings of the 46th IEEE Frequency Control Symposium, 465-469, 1992.
- 4. Vig, J.R., "Quartz Crystal Resonators and Oscillators for Frequency Control and Timing Applications," SLCET-TR-88-1 (Rev. 5.2), US Army LABCOM, Fort Monmouth, 1992.
- Suter, J.J., R.H. Maurer, and J.D. Kinnison, "The Susceptibility of Electrodeless Quartz Crystal BVA Resonators to Proton Ionization Effects," IEEE Trans. Nucl. Sci., 35, 1451-1455, 1988.
- Suter, J.J., and R.H. Maurer, "Low and High Dose Photon Irradiation of Quartz Crystal Resonators," Proceedings of the 17th Annual Precise Time and Time Interval (PTTI) Meeting, 393-403, 1985.
- Suter, J.J., and R.H. Maurer, "Low and Medium Dose Radiation Sensitivity of Quartz Crystal Resonators with Different Aluminum Impurity Content," IEEE Trans. Ultrason., Ferroelectrics, and Freq. Control, UFFC-34, 667-673, 1987.
- Norton, J.R., J.M. Cloeren, and J.J. Suter, "Results from Gamma Ray and Proton Beam Radiation Testing of Quartz Resonators," IEEE Trans. Nucl. Sci., 31, 1230-1235, 1984.
- Flanagan, T.M., R.E. Leadon, and D.L. Shannon, "Evaluation of Mechanisms for Low-Dose Frequency Shifts in Crystal Oscillators," IEEE Trans. Nucl. Sci., 33, 1447-1453, 1986.
- Cash, P.E., D.A. Emmons, and W. Stapor, "Low Dose Proton Radiation Sensitivity of Quartz Resonators," Proceedings of the 10th European Forum on Time and Frequency, 10-20, 1996.
- Sigel, G.H., Jr., E.J. Friebele, R.J. Ginther, and D.L. Griscom, "Effects of Stoichiometry on the Radiation Response of SiO2," IEEE Trans. Nucl. Sci., 21, 56-61, 1974.
- Bahadur, H., "Low-temperature Irradiation Effects in Natural Crystalline Quartz," J. Appl. Phys., 75, 1420-1425, 1994.
- Revesz, A.G., "Defect Structure and Irradiation Behavior of Noncrystalline SiO2," IEEE Trans. Nucl. Sci., 18, 113-116, 1972.
- Allan, D.W., "Statistics of Atomic Frequency Standards," Proceedings of the IEEE, 34, 221-230, 1966.

TECHNOLOGY OPERATIONS

The Aerospace Corporation functions as an "architect-engineer" for national security programs, specializing in advanced military space systems. The Corporation's Technology Operations supports the effective and timely development and operation of national security systems through scientific research and the application of advanced technology. Vital to the success of the Corporation is the technical staff's wide-ranging expertise and its ability to stay abreast of new technological developments and program support issues associated with rapidly evolving space systems. Contributing capabilities are provided by these individual Technology Centers:

Electronics Technology Center: Microelectronics, VLSI reliability, failure analysis, solid-state device physics, compound semiconductors, radiation effects, infrared and CCD detector devices, Micro-Electro-Mechanical Systems (MEMS), and data storage and display technologies; lasers and electro-optics, solid state laser design, micro-optics, optical communications, and fiber optic sensors; atomic frequency standards, applied laser spectroscopy, laser chemistry, atmospheric propagation and beam control, LIDAR/LADAR remote sensing; solar cell and array testing and evaluation, battery electrochemistry, battery testing and evaluation.

Mechanics and Materials Technology Center: Evaluation and characterization of new materials: metals, alloys, ceramics, polymers and composites; development and analysis of advanced materials processing and deposition techniques; nondestructive evaluation, component failure analysis and reliability; fracture mechanics and stress corrosion; analysis and evaluation of materials at cryogenic and elevated temperatures; launch vehicle fluid mechanics, heat transfer and flight dynamics; aerothermodynamics; chemical and electric propulsion; environmental chemistry; combustion processes; spacecraft structural mechanics, space environment effects on materials, hardening and vulnerability assessment; contamination, thermal and structural control; lubrication and surface phenomena; microengineering technology and microinstrument development.

Space and Environment Technology Center: Magnetospheric, auroral and cosmic ray physics, wave-particle interactions, magnetospheric plasma waves; atmospheric and ionospheric physics, density and composition of the upper atmosphere, remote sensing, hyperspectral imagery; solar physics, infrared astronomy, infrared signature analysis; effects of solar activity, magnetic storms and nuclear explosions on the earth's atmosphere, ionosphere and magnetosphere; effects of electromagnetic and particulate radiations on space systems; component testing, space instrumentation; environmental monitoring, trace detection; atmospheric chemical reactions, atmospheric optics, light scattering, state-specific chemical reactions and radiative signatures of missile plumes, and sensor out-of-field-of-view rejection.